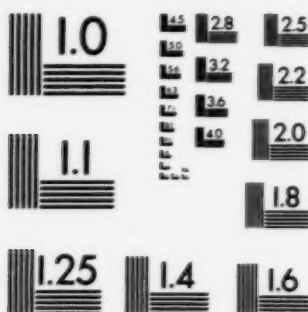


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for Mineral  
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de la technologie  
des minéraux  
et de l'énergie

### THE DETERMINATION OF MAJOR AND MINOR ELEMENTS IN THE ASH OF SOLID MINERAL FUELS (Method by Flame Atomic Absorption Spectrometry)

R. Prokopuk and L. Janke  
Fifth Working Draft  
ISO WG 13/TC27  
REPLACES ISO TC27 N1340

May 1983

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## APPENDIX



## 1. SCOPE AND FIELD OF APPLICATION

This proposal specifies the procedure for the elemental analysis of twelve major and minor constituents in ash derived from either, solid mineral fuels or semi-coke and coke residues obtained from Brown coals and lignites.

Table 1 identifies elements of interest and their applicable concentration ranges (Note 1).

Table 1 - Concentration ranges

| Element   | Reported as oxide       | Range per cent |
|-----------|-------------------------|----------------|
| Aluminum  | $\text{Al}_2\text{O}_3$ | 1.0-70         |
| Barium    | $\text{BaO}$            | 0.01-5         |
| Calcium   | $\text{CaO}$            | 0.05-60        |
| Iron      | $\text{Fe}_2\text{O}_3$ | 0.05-80        |
| Magnesium | $\text{MgO}$            | 0.05-35        |
| Manganese | $\text{Mn}_3\text{O}_4$ | 0.005-1.0      |
| Potassium | $\text{K}_2\text{O}$    | 0.01-8.0       |
| Silicon   | $\text{SiO}_2$          | 1.0-90         |
| Sodium    | $\text{Na}_2\text{O}$   | 0.05-15        |
| Strontium | $\text{SrO}$            | 0.01-2.0       |
| Titanium  | $\text{TiO}_2$          | 0.01-10.0      |
| Zinc      | $\text{ZnO}$            | 0.01-1.0       |

Note 1: Both sulphur and phosphorus exist in ashes derived from solid mineral fuels and semi-coke or coke residues of Brown coals and lignites. These elements may be determined as phosphorus pentoxide ( $\text{P}_2\text{O}_5$ ) using ISO 622 and sulphur trioxide ( $\text{SO}_3$ ) using ISO/TC 27N 1341 and included in material balance, when reporting.

## 2. REFERENCES

ISO 1988, Hard Coal Sampling.

ISO 1171, Solid Mineral Fuels - The Determination of Ash.

ISO TC 27N 1341, The Determination of Major Elements in the Ash of Solid Mineral Fuels (Classical and Instrumental Methods, Fourth Draft, September, 1982).

## 3. SUMMARY OF METHOD

Ash, prepared in accordance with ISO 1171 and ground in an agate mortar to pass a 0.063 mm square mesh test sieve, is fused with either lithium metaborate or lithium tetraborate (Note 2), followed by final dissolution of the melt in dilute nitric acid. The elements listed in Table 1 are quantified, utilizing flame atomic absorption spectroscopic techniques against calibration standards containing a matching solution matrix.

Note 2: Lithium metaborate is a recommended flux for the decomposition of highly siliceous ashes. Lithium tetraborate is preferred for ashes containing high contents of iron oxides.

## 4. APPARATUS

### 4.1 Furnaces

4.1.1 Ashing furnace as specified in ISO 1171

4.1.2 Fusion furnace with an operating temperature range of 1000  $\pm$ 50°C;  
or Mekker Burners

### 4.2 Crucibles and Lids

Crucibles and lids for fusion of the ashes shall be made of pure platinum or platinum/gold alloy (95/5 wt %) with a capacity of at least 30 mL.

#### 4.3 Atomic Absorption Spectrophotometer

The instrument should be equipped with interchangeable air/acetylene and nitrous oxide/acetylene flame burners and either absorbance or direct concentration readouts. The fuel gas composition and wavelengths listed in Table 2 are recommended. Other instrument parameters shall be optimized in accordance with manufacturers instructions.

#### 4.4 Hotplate

Hotplate with an operating range to 300°C and magnetic stirring capabilities is recommended; or a sand bath.

Table 2 - Atomic absorption spectroscopy

| Element   | Wavelength<br>nanometer | Fuel<br>composition | Limitations   |
|-----------|-------------------------|---------------------|---------------|
| Aluminum  | 309.3                   | $N_2O/C_2H_2$       |               |
| Barium    | 553.6                   | $N_2O/C_2H_2$       |               |
| Calcium   | 422.7                   | $N_2O/C_2H_2$       |               |
| Iron      | 248.3                   | $N_2O/C_2H_2$       |               |
| Magnesium | 285.2                   | $N_2O/C_2H_2$       |               |
| Manganese | 279.5                   | $N_2O/C_2H_5$       |               |
| Potassium | 766.5                   | Air/ $C_2H_2$       | <2.5% $K_2O$  |
|           | 769.5                   | Air/ $C_2H_2$       | >2.5% $K_2O$  |
| Silicon   | 251.6                   | $N_2O/C_2H_2$       |               |
| Sodium    | 589.0                   | Air/ $C_2H_2$       | <2.5% $Na_2O$ |
|           | 589.6                   | Air/ $C_2H_2$       | >2.5% $Na_2O$ |
| Strontium | 460.7                   | $N_2O/C_2H_2$       |               |
| Titanium  | 365.3                   | $N_2O/C_2H_2$       |               |
| Zinc      | 213.9                   | Air/ $C_2H_2$       |               |

#### 4.5 Storage Bottles

Bottles composed of polyethylene or polypropylene with screw caps and an air tight seal are recommended for solution storage (Note 3).

#### 4.6 Glassware

4.6.1 Grade A volumetric glassware shall be used, unless otherwise specified (Note 3).

Note 3: All bottles and glassware shall be conditioned by presoaking in dilute nitric acid (5.4.4) for at least 24 h prior to usage.

4.6.2 Grade A beakers

4.6.3 Grade A pipettes

### 5. REAGENTS

#### 5.1 General

Chemicals of analytical grade purity conforming to national standards shall be used in all tests.

#### 5.2 Water

Distilled water or its equivalent, shall be used in the preparation of all solutions.

#### 5.3 Fluxing Agent

Lithium tetraborate ( $\text{Li}_2\text{B}_4\text{O}_7$ ) or lithium metaborate ( $\text{LiBO}_2$ ) with a minimum purity of 98%.

#### 5.4 Solutions

5.4.1 Nitric acid ( $\rho_{20}$  1420 kg per  $\text{m}^3$ )

5.4.2 Nitric acid (1+1), dilute 1 part nitric acid (5.4.1) with 1 part water (5.2).

5.4.3 Nitric acid (1+9), dilute 1 part nitric acid (5.4.1) with 9 parts water (5.2).

5.4.4 Nitric acid (1+19), dilute 1 part nitric acid (5.4.1) with 19 parts water (5.2).

5.4.5 Hydrochloric acid ( $\rho_{20}$  1190 kg per  $\text{m}^3$ ).

5.4.6 Hydrochloric acid (1+1), dilute 1 part hydrochloric acid (5.4.5) with 1 part water (5.2).

## 5.5 Standard Stock Solutions

5.5.1 Standard aluminum solution (4000 mg per L  $\text{Al}_2\text{O}_3$ )

Add 40 mL of hydrochloric acid (5.4.5) to 40 mL of water (5.2) containing  $2.1170 \pm 0.005$  g of freshly prepared aluminum metal (5.1). Heat to just below boiling for approximately 5 min. Cool to ambient and add 20 mL of nitric acid (5.4.2). Cover the beaker with a watchglass and reheat until all the aluminum dissolves. Cool to ambient and transfer quantitatively to a 1 L volumetric flask. Dilute to the mark with water (5.2).

5.5.2 Standard barium solution (400 mg per L BaO)

Dissolve  $0.5148 \pm 0.0005$  g of predried barium carbonate ( $150^\circ\text{C}$  for 2 h) in 40 mL of water (5.2). Add slowly 20 mL of nitric acid (5.4.2). Heat and agitate to degas carbon dioxide bubbles. Add slowly a further 80 mL nitric acid (5.4.2). Cool to ambient and transfer quantitatively to a 1 L volumetric flask. Dilute to the mark with water (5.2).

5.5.3 Standard calcium solution (200 mg per L CaO)

Dissolve  $0.3570 \pm 0.0005$  g of predried calcium carbonate ( $200^\circ\text{C}$  for 1 h) in 150 mL of water (5.2). Add slowly 20 mL of nitric acid (5.4.2). Heat and agitate to degas carbon dioxide bubbles. Add slowly, a further 80 mL nitric acid (5.4.2). Cool to ambient and transfer quantitatively to a 1 L volumetric flask. Dilute to the mark with water (5.2).

5.5.4 Standard iron solution (1000 mg per L  $\text{Fe}_2\text{O}_3$ )

Dissolve  $0.6995 \pm 0.0005$  g of freshly prepared iron metal in 100 mL of nitric acid (5.4.2). Add 5 mL of hydrochloric acid (5.4.6). When dissolution is complete, cool to ambient and transfer quantitatively to a 1 L volumetric flask. Dilute to the mark with water (5.2).

#### 5.5.5 Standard magnesium solution (200 mg per L MgO)

Dissolve  $0.2000 \pm 0.0005$  g of preignited magnesium oxide ( $1000^{\circ}\text{C}$  for 30 min) in 100 mL of nitric acid (5.4.2). When dissolution is complete, cool to ambient and transfer quantitatively to a 1 L volumetric flask. Dilute to the mark with water (5.2).

#### 5.5.6 Standard manganese solution (400 mg per L $\text{Mn}_3\text{O}_4$ )

Dissolve  $0.2882 \pm 0.0005$  g of freshly prepared manganese metal in 100 mL of nitric acid (5.4.2). When dissolution is complete, cool to ambient and transfer quantitatively to a 1 L volumetric flask. Dilute to the mark with water (5.2).

#### 5.5.7 Standard potassium solution (200 mg per L $\text{K}_2\text{O}$ )

Dissolve  $0.3166 \pm 0.0005$  g of predried potassium chloride ( $110^{\circ}\text{C}$  for 1 h) in 200 mL of water (5.2). Add 100 mL of nitric acid (5.4.2) and transfer quantitatively to a 1 L volumetric flask. Dilute to the mark with water (5.2).

#### 5.5.8 Standard silicon solution (2000 mg per L $\text{SiO}_2$ )

Weight  $1.0000 \pm 0.0001$  g of pure preignited silica ( $1000^{\circ}\text{C}$  for 1 h) into a platinum or platinum/gold crucible (4.2). Add 5 g of flux (5.3) and mix thoroughly. Place crucible and sample in furnace (4.1.2) and fuse at  $1000 \pm 50^{\circ}\text{C}$  until a clear melt is obtained (Note 4).

Note 4: In order to hasten fusion process, the pure silica should be ground to pass a 80 micron test sieve and the fusion mixture should be swirled frequently.

Pour the melt into a 400 mL borosilicate beaker containing 250 mL of nitric acid (5.4.2). Add a PTFE coated magnetic stirring bar to the beaker. Cool the crucible to ambient. Clean the crucibles exterior surfaces before placing in the beaker. Cover the beaker with a watchglass before placing on magnetic stirring hot plate (4.4). While heating slowly to just below boiling point, stir the solution vigorously (Note 5).

Note 5: If stirring motion is too slow or heating rate too fast, localized high silicon concentrations may cause precipitation, invalidating preparation.

When dissolution of fused bead is complete, remove the crucible using precleaned platinum-tipped tweezers. Wash the crucible with water (5.2), collecting the washings in the beaker. Cool the solution to ambient and transfer quantitatively to a 500 mL volumetric flask. Dilute to the mark with water (5.2).

5.5.9 Standard sodium solution (200 mg per L  $\text{Na}_2\text{O}$ )

Dissolve  $0.3772 \pm 0.0005$  g of predried sodium chloride ( $110^\circ\text{C}$  for 1 h) in 50 mL of water (5.2). Add 100 mL of nitric acid (5.4.2) and transfer quantitatively to a 1 L volumetric flask. Dilute to the mark with water (5.2).

5.5.10 Standard strontium solution (200 mg per L  $\text{SrO}$ )

Dissolve  $0.2849 \pm 0.0005$  g of predried strontium carbonate ( $110^\circ\text{C}$  for 1 h) in 50 mL of water (5.2). Add slowly 100 mL nitric acid (5.4.2). Heat and agitate to degas carbon dioxide bubbles. Cool to ambient and transfer quantitatively to a 1 L volumetric flask. Dilute to the mark with water (5.2).

5.5.11 Standard titanium solution (400 mg per L  $\text{TiO}_2$ )

Dissolve  $0.2398 \pm 0.0005$  g of freshly prepared titanium metal (5.1) in 50 mL of hydrochloric acid (5.4.6). Cover the beaker with a watchglass and heat on a sand bath (4.4) to dissolve. When dissolution is complete, cool to ambient and transfer quantitatively to 1 L volumetric flask. Dilute to the mark with water (5.2).

5.5.12 Standard zinc solution (200 mg per L  $\text{ZnO}$ )

Dissolve  $0.1607 \pm 0.0005$  g of freshly prepared zinc metal in 100 mL of nitric acid (5.4.2). When dissolution is complete, cool to ambient and transfer quantitatively to a 1 L volumetric flask. Dilute to the mark with water (5.2).

#### 5.5.13 Standard lanthanum/zirconium solution (20 g La and 10 g Zr per L)

Dissolve  $23.455 \pm 0.001$  g of preignited lanthanum oxide ( $1000^\circ\text{C}$  for 1 h) and  $35.328 \pm 0.001$  g of predried ( $110^\circ\text{C}$  for 1 h) zirconium oxychloride ( $\text{ZrOCl}_2 \cdot 8\text{H}_2\text{O}$ ) in 100 mL of nitric acid (5.4.2) (Note 6). Cool to ambient and transfer quantitatively to a 1 L volumetric flask. Dilute to the mark with water (5.2).

Note 6: Lanthanum serves as an ionization suppressant while zirconium is added as a buffer to eliminate the interference of aluminum to the analysis of titanium and vice versa.

#### 5.5.14 Flux solution (20 g Li per L)

Dissolve  $20.000 \pm 0.001$  g of fused lithium tetraborate or metaborate (5.3) in 500 mL of nitric acid (5.4.2). Transfer quantitatively to a 1 L volumetric flask and dilute to the mark with water (5.2).

#### 5.5.15 Matrix matching solution

Dilute 200 mL of flux solution (5.5.14) with 100 mL of the standard lanthanum/zirconium solution (5.5.13). Transfer quantitatively to a 1 L volumetric flask. Dilute to the mark with nitric acid (5.4.2).

### 5.6 Calibration Solutions

Prepare calibration solutions in 250 mL volumetric flasks in accordance with Table 3. Ensure homogeneity of each solution by thoroughly mixing.

Table 3 - Calibration solution preparation

| Standard stock<br>solution  | Calibration solution number |      |     |      |     |      |      |      |
|-----------------------------|-----------------------------|------|-----|------|-----|------|------|------|
|                             | 1                           | 2    | 3   | 4    | 5   | 6    | 7    | 8    |
|                             | Volume added mL             |      |     |      |     |      |      |      |
| Aluminum (5.5.1)            | 0                           | 0.25 | 0.5 | 1.25 | 2.5 | 5.0  | 7.5  | 10.0 |
| Barium (5.5.2)              | 0                           | 0.5  | 1.0 | 2.5  | 5.0 | 7.5  | 10.0 | 12.5 |
| Calcium (5.5.3)             | 0                           | 0.5  | 1.0 | 2.5  | 5.0 | 10.0 | 15   | 20   |
| Iron (5.5.4)                | 0                           | 0.5  | 1.0 | 2.5  | 5.0 | 10.0 | 15   | 20   |
| Magnesium (5.5.5)           | 0                           | 0.5  | 1.0 | 2.5  | 5.0 | 7.5  | 10.0 | 20   |
| Manganese (5.5.6)           | 0                           | 0.5  | 1.0 | 2.5  | 5.0 | 7.5  | 10.0 | 12.5 |
| Potassium (5.5.7)           | 0                           | 0.5  | 1.0 | 2.5  | 5.0 | 10.0 | 15   | 20   |
| Silicon (5.5.8)             | 0                           | 80   | 60  | 40   | 20  | 10.0 | 5.0  | 2.0  |
| Sodium (5.5.9)              | 0                           | 0.5  | 1.0 | 2.5  | 5.0 | 10.0 | 15   | 20   |
| Strontium (5.5.10)          | 0                           | 0.25 | 0.5 | 1.0  | 2.0 | 3.0  | 4.0  | 5.0  |
| Titanium (5.5.11)           | 0                           | 0.5  | 1.0 | 2.5  | 5.0 | 7.5  | 10.0 | 12.5 |
| Zinc (5.5.12)               | 0                           | 0.25 | 0.5 | 1.0  | 2.0 | 3.0  | 4.0  | 5.0  |
| Flux (5.5.14)               | 50                          | 10   | 20  | 30   | 40  | 45   | 50   | 50   |
| Matrix Matching<br>(5.5.15) | 25                          | 25   | 25  | 25   | 25  | 25   | 25   | 25   |

## 6. PREPARATION OF SAMPLE

### 6.1 Ash General

Ash samples shall be prepared in accordance with ISO 1171 (Note 7,8). Grind the incinerated ash in an agate mortar to pass a 0.063 mm square mesh test sieve. Store samples in an airtight container.

Note 7: Ash samples removed from industrial hearths are considered acceptable provided they are pretreated as described in 6.1.

Note 8: Some samples lose the more volatile constituents, i.e., sodium, when incinerated at conditions outlined in ISO 1171. To avoid risk of losses, samples rich in volatile elements, may be incinerated at 500°C. The deviated ashing temperature and new ash content at this ashing temperature must be noted when reporting results.

## 6.2 Loss in Mass on Heating

### 6.2.1 Procedure

Weigh 1.000  $\pm$  0.2 g of ash (6.1) into a platinum or platinum/gold crucible (4.2). Incinerate sample and crucible in furnace (4.1.2) at 815  $\pm$  10°C, until the difference between the results of two weighings is less than 0.004 g.

### 6.2.2 Calculation

The loss in mass on heating, X, as a percentage is calculated from the formula:

$$X = \frac{M_0 - M_1}{M_0} \times 100$$

where

$M_0$  - the initial weight of ash used, g

$M_1$  - the final weight of ash remaining in crucible, g

## 7. PROCEDURE

### 7.1 General

Analysis must be completed in duplicate on each sample using two separate test portions of the ash (6.1).

### 7.2 Control Blank

A blank consisting of 1.00 g of flux (5.3) shall be subjected to entire procedure - fusion (7.4), dissolution (7.6), and instrumental measurement (7.7) for background correction purposes.

### 7.3 Quality Control

A certified reference material should be treated in parallel with the analysis of the test sample and under the same conditions (Note 9).

Note 9: Suitable materials include ES681-1, NBS 1632a, NBS 1633a, BCS269, etc.

#### 7.4 Sample Fusion

Weigh  $0.1000 \pm 0.0002$  g of prepared sample (6.1) into a platinum or platinum/gold crucible (4.2). Add 0.50 g of flux (5.3) and mix thoroughly. Add an additional 0.50 g flux (5.3) to blanket the mixture. Place the crucible onto a clean silica or refractory support tray before inserting into a furnace (4.1.2) preheated at  $1000 \pm 50^\circ\text{C}$ . Incinerate for 15 min (Note 10).

Note 10: This time interval is usually sufficient to fuse the mixture completely. If the time period is insufficient, use discretion, but do not exceed 1 h.

Cool the tray and the crucible to ambient. Carefully, rinse with water (5.2), the bottom and outside surfaces of the crucible. Discard these washings.

#### 7.5 Sample Dissolution

Place the cleaned crucible containing the fused sample into a 400 mL beaker in an upright position. Place a clean PTFE coated magnetic stirring bar in the crucible. Add at least 125 mL of nitric acid (5.4.2) to immerse totally, the crucible. Heat the solution with constant stirring on a hotplate to just below the boiling point and maintain this condition for not more than 30 min (Note 11).

Note 11: The time and temperature selected is usually sufficient to dissolve the melt completely. If stirring is not maintained constantly, a precipitate may form invalidating the procedure and the sample preparation must be repeated.

Remove the beaker containing the crucible from the hotplate and cool to ambient. Quantitatively transfer the solution to a 250 mL volumetric flask. Wash with water (5.2), the crucible surfaces and the beaker and transfer the washings to volumetric flask. Add 25 mL of standard lanthanum/zirconium solution (5.5.13). Dilute to mark with nitric acid (5.4.2) (Note 12).

Note 12: This solution contains 400 mg per L of solids with respect to total sample, 4 g per L of fluxing agent (5.3) and matrix matching solution (5.5.15).

## 7.6 Sample Test Solutions

### 7.6.1 Test solution A

The stock sample solution prepared in Clause 7.5 is so designated.

### 7.6.2 Test solution B

Pipette 10.0 mL of test solution A (7.6.1) into a 50 mL volumetric flask. Dilute to mark with matrix matching solution (5.5.15).

### 7.6.3 Test solution C

Pipette 10.0 mL of test solution A (7.6.1) into a 100 mL volumetric flask. Dilute to mark with matrix matching solution (5.5.15).

### 7.6.4 Test solution D

Pipette 5.0 mL of test solution A (7.6.1) into a 100 mL volumetric flask. Dilute to mark with matrix matching solution (5.5.15).

## 7.7 Instrumental Measurement

### 7.7.1 General

Measure the absorbance values of the calibration standards (5.6) and sample test solutions (7.6) (Note 13). Ensure optimum flame and electronic spectrophotometer conditions are used, as recommended by the manufacturer.

Note 13: Periodically rerun calibration standards (7.6) in order to monitor instrument drift and the prepared reference sample (7.3) for accuracy assessment.

### 7.7.2 Calibration graphs

Prepare calibration graphs plotting absorbance reading observed for working calibration standards against percentage of oxides in the standards as shown in Table 4 (Note 14).

Note 14: Alternately, if the atomic absorption spectrophotometer uses direct concentration readout, graphs are unnecessary.

Table 4 - Working calibration standards

| Oxide                   | Sample test solution | Calibration solution number |       |       |      |      |      |      |      |
|-------------------------|----------------------|-----------------------------|-------|-------|------|------|------|------|------|
|                         |                      | 1                           | 2     | 3     | 4    | 5    | 6    | 7    | 8    |
|                         |                      | Concentration per cent      |       |       |      |      |      |      |      |
| $\text{Al}_2\text{O}_3$ | A                    | 0                           | 0.5   | 1.0   | 2.5  | 5.0  | 10.0 | 15   | 20   |
|                         | B                    | 0                           | 2.5   | 5.0   | 12.5 | 25   | 50   | 75   | 100  |
| BaO                     | A                    | 0                           | 0.1   | 0.2   | 0.5  | 1.0  | 1.5  | 2.0  | 2.5  |
|                         | B                    | 0                           | 0.5   | 1.0   | 2.5  | 5.0  | 7.5  | 10.0 | 12.5 |
| CaO                     | A                    | 0                           | 0.05  | 0.1   | 0.25 | 0.5  | 1.0  | 1.5  | 2.0  |
|                         | B                    | 0                           | 0.25  | 0.5   | 1.25 | 2.5  | 5.0  | 7.5  | 10.0 |
|                         | C                    | 0                           | 0.5   | 1.0   | 2.5  | 5.0  | 10.0 | 15   | 20   |
|                         | D                    | 0                           | 1.0   | 2.0   | 5.0  | 10.0 | 20   | 30   | 40   |
| $\text{Fe}_2\text{O}_3$ | A                    | 0                           | 0.25  | 0.5   | 1.25 | 2.5  | 5.0  | 7.5  | 10.0 |
|                         | B                    | 0                           | 1.25  | 2.5   | 6.25 | 12.5 | 25   | 37.5 | 50   |
|                         | C                    | 0                           | 2.5   | 5.0   | 13   | 25   | 50   | 75   | 100  |
| MgO                     | A                    | 0                           | 0.05  | 0.1   | 0.25 | 0.5  | 1.0  | 1.5  | 2.0  |
|                         | B                    | 0                           | 0.25  | 0.5   | 1.25 | 2.5  | 5.0  | 7.5  | 10.0 |
|                         | C                    | 0                           | 0.5   | 1.0   | 2.5  | 5.0  | 10.0 | 15   | 20   |
|                         | D                    | 0                           | 1.0   | 2.0   | 5.0  | 10.0 | 20   | 30   | 40   |
| $\text{Mn}_3\text{O}_4$ | A                    | 0                           | 0.1   | 0.2   | 0.5  | 1.0  | 1.5  | 2.0  | 2.5  |
| $\text{K}_2\text{O}$    | A                    | 0                           | 0.05  | 0.1   | 0.25 | 0.5  | 1.0  | 1.5  | 2.0  |
|                         | B                    | 0                           | 0.25  | 0.5   | 1.25 | 2.5  | 5.0  | 7.5  | 10.0 |
| $\text{SiO}_2$          | A                    | 0                           | 80    | 60    | 40   | 20   | 10.0 | 5.0  | 2.0  |
|                         | B                    | 0                           | -     | -     | -    | 100  | 50   | 25   | 10.0 |
| $\text{Na}_2\text{O}$   | A                    | 0                           | 0.05  | 0.1   | 0.25 | 0.5  | 1.0  | 1.5  | 2.0  |
|                         | B                    | 0                           | 0.25  | 0.5   | 1.25 | 2.5  | 5.0  | 7.5  | 10.0 |
|                         | C                    | 0                           | 0.5   | 1.0   | 2.5  | 5.0  | 10.0 | 15   | 20   |
| SrO                     | A                    | 0                           | 0.025 | 0.05  | 0.1  | 0.2  | 0.3  | 0.4  | 0.5  |
|                         | B                    | 0                           | 0.125 | 0.25  | 0.5  | 1.0  | 1.5  | 2.0  | 2.5  |
| $\text{TiO}_2$          | A                    | 0                           | 0.1   | 0.2   | 0.5  | 1.0  | 1.5  | 2.0  | 2.5  |
|                         | B                    | 0                           | 0.5   | 1.0   | 2.5  | 5.0  | 75   | 10.0 | 12.5 |
| ZnO                     | A                    | 0                           | 0.025 | 0.05  | 0.1  | 0.2  | 0.3  | 0.4  | 0.5  |
|                         | B                    | 0                           | 0.125 | 0.025 | 0.5  | 1.0  | 1.5  | 2.0  | 2.5  |

### 7.7.3 Curve linearity

The observed difference of either the absorbance or direct concentration readout between the blank solution (7.2) and the highest concentration calibration solution from which a calibration curve is constructed, should exceed 1.7 times the observed difference at mid range. If the value exceeds limit, identify and correct source of error.

### 7.7.4 Background Correction

Use of the matrix matching solution (5.5.15) technique for the ranges specified in Table 1, reduces direct and indirect interferences to tolerable levels. Therefore, background correction may be unnecessary.

## 8. CALCULATION AND EXPRESSION OF RESULTS

### 8.1 Calculations

The mean concentration between duplicate analyzed using two separate test portions (7.1), shall be reported on a dry basis, as percentage of oxides in the ash. These results are then corrected to a dry combustible-free basis (Note 15) using the following formula:

$$\text{Dry combustible free basis (per cent)} = \frac{100 \times \text{oxide (per cent)}}{100 - [\text{loss on mass heating (per cent)}]}$$

Note 15: A material balance corrected to a dry combustible-free basis and including sulphur trioxide, phosphorous pentoxide, major and minor elements should range from 98 to 102 per cent accountability.

### 8.2 Precision

Table 5 - Permissible deviation limits

APPENDIX A

